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ENGINEERING PROPERTY DATA ON ROCKET PROPELLANTS

Fourth Quarterly Report

Rocketdyne
A Division of North American Aviation, Inc.
6633 Canoga Avenue
Canoga Park, California

July 1967

Group 4
Downgraded at 3-Year Intervals
Declassified After 12 Years

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Air Force Rocket Propulsion Laboratory
Research and Technology Division
Edwards Air Force Base, California
Air Force Systems Command
United States Air Force

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FOREWORD

(U) This is a quarterly technical report submitted under G.O. 07119 in compliance with Contract AF04(611)-11407. The research reported herein, which covers the period 1 April through 30 June 1967, was sponsored by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, United States Air Force, Edwards, California, with Mr. G. Allen Beale acting as the Air Force Project Officer.

(U) This program was conducted in the Chemical and Material Sciences Department of the Rocketdyne Research Division, with Dr. E. F. C. Cain serving as Program Manager and Mr. M. T. Constantine serving as the Responsible Project Scientist. Technical personnel who have contributed to this effort include K. J. Youel (Phases I and III), Dr. J. F. Hon (Phase II), Dr. W. Unterberg (Phase II), Dr. S. E. Rodriguez (Phase II), J. V. Lecce (Phase II), R. W. Melvold (Phase II), J. Quaglino (Phase II), and M. M. Williams (Phase III).

(U) This report has been assigned the Rocketdyne identification number R-6638-4.

(U) Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

W. H. EBELKE, Colonel, USAF
Chief, Propellant Division

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ABSTRACT

(U) The results of the fifth quarter year's effort of a 24-month program on the analytical and experimental characterization of the physical properties of selected liquid propellants are presented in three phases. In Phase I, a continuous review of the literature was conducted to ensure the acquisition and documentation of the latest possible propellant properties data for evaluation and possible inclusion into a propellant properties handbook. Phase II experimental efforts have resulted in the measurement of chlorine trifluoride vapor pressure in the high-temperature region and an estimation of critical properties; sonic velocity in chlorine trifluoride and chlorine pentafluoride under pressurized conditions; nitrogen gas solubility in chlorine pentafluoride, specific heat of saturated liquid chlorine trifluoride; thermal conductivity of MHF-5; and viscosity of chlorine pentafluoride. Phase III efforts included the evaluation and assembly of all data generated in Phases I and II, a curve fit of specific heat data on solid chlorine trifluoride, and a report of unpublished data on the normal boiling point of chlorine pentafluoride and chlorine pentafluoride storability.

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INTRODUCTION

(U) Under Contract AF04(611)-10546, Rocketdyne completed a 12-month analytical and experimental program (Ref. 1) on the rational and systematic physical characterization of selected liquid rocket propellants. The overall objective of this program, which was conceived as an initial step toward elimination of propellant data gaps, was to assemble and experimentally complete the data on essential physical properties of current and near-term liquid rocket propellants over temperature and pressure ranges of practical use to propulsion engineering. During this program, experimental efforts resulted in measurement of: (1) chlorine trifluoride (ClF_3) phase properties (including critical temperature); (2) IRFNA and chlorine pentafluoride (ClF_5) sonic velocities (and calculation of compressibilities); (3) ClF_3 and $CH_3N_2H_3$ specific heats (and correction of previously determined ClF_5 specific heat data); (4) thermal conductivities of the 50 N_2H_4 - 50 $(CH_3)_2N_2H_2$ fuel blend and $CH_3N_2H_3$; and (5) the design and preliminary assembly of apparatuses for measurement of inert-gas solubility in liquids and liquid viscosities at extended temperatures and pressures. Analytical efforts, initiated with an extensive literature survey, included the assembly and evaluation of physical property data on MHF-1, MHF-3, MHF-5, ClF_3, and ClF_5 for future correlation and summary publication.

(U) The present three-phase program, being conducted under Contract AF04(611)-11407, represents a 24-month extension and expansion of the objectives of the previous effort. Phase I effort consists of a continuous review of the current literature to document reported propellant properties. In Phase II, effort is directed at the experimental determination of unavailable engineering data, for selected oxidizers and fuels, which are required to design rocket engine hardware. Effort under Phase III includes the compilation, correlation, and evaluation of all data obtained from Phases I and II and presentation of the valid data in an annual technical report.

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During the first year of the current program, which has been reported in an Annual Summary Report (Ref. 2), the continuous documentation of current propellant properties data was maintained with a detailed review of all reports and of all current releases of the standard abstract sources. Experimental efforts in this 12-month period resulted in measurements of (1) ClF_5 density and vapor pressure (to extend the available data [illegible]), (2) saturated liquid ClF_3 and ClF_5 sonic velocities (and [illegible] of adiabatic compressibilities); (3) $N_{2(g)}$ solubility [illegible]; (4) UDMH, 50 N_2H_4 - 50 $(CH_3)_2N_2H_2$ and MHF-3 specific heats, (5) thermal conductivities of UDMH, MHF-[illegible] and MHF-5; and (6) ClF_5 viscosity. Phase III analytical efforts resulted in the correlation of Phase II experimental data, the preparation [illegible] complete physical property bibliographies on B_2H_6 and N_2H_4 and the initiation of an N_2O_4 physical property bibliography, and the publication of N_2H_4 physical property data sheets.

(U) Progress achieved in a continuation of these objectives and efforts during the fifth quarter year of the current program is summarized in this report.

SUMMARY

(U) Analytical and experimental research conducted during the fifth quarter year of a current 24-month program to complete the data on essential physical properties of current and near-term liquid propellants of interest to the Air Force is described in three phases.

(U) In Phase I, a continuous survey of the current propellant literature has resulted in the preliminary screening of 2279 reports acquired and cataloged by Rocketdyne during this period; 118 of these reports were reviewed in detail for pertinent engineering properties data on liquid propellants.

(U) The experimental characterization of essential physical properties of selected propellants was conducted under Phase II. During the current report period, these efforts were directed at measurements of ClF_3 phase properties in the critical region, sonic velocity in ClF_5 and ClF_3 under pressurized conditions, gaseous nitrogen solubility in ClF_5, ClF_3 and MHF-3 specific heat, MHF-5 thermal conductivity, and ClF_5 viscosity.

(U) A constant-volume vapor pressure apparatus was used to extend the available experimental data on ClF_3 phase properties into the critical range. As a result of the current measurements, the vapor pressure of ClF_3 from 26 to 149 C (77 to 299 F) was correlated with the following equations:

$$\log P_{(psia)} = 5.9929 - \frac{2453.7}{T_{(R)}}$$

and

$$\log P_{(atm)} = 4.8257 - \frac{1363.1}{T_{(K)}}$$

These data were used with a previously determined critical temperature of 170.6 ±0.5 C ([illegible]) to calculate a critical pressure of 961 psia (65.4 atm), and with previously determined density data to estimate a critical density of 0.88 gm/cc (54.9 lb/cu ft) and a specific volume of 1.14 cc/gm (0.018 cu ft/lb).

(U) Measurements of sonic velocity in liquid ClF_5 and ClF_3 were conducted under total pressures (with gaseous nitrogen) of 500, 800, and 1000 psia. These data, which are presently being completed and correlated, substantiate the normally predicted behavior of sonic velocity in pressurized liquids (i.e., increase with an increase in pressure).

(U) Using an improved experimental technique, measurements of gaseous nitrogen solubility in liquid ClF_5 were redetermined. The resulting solubilities of 1.83×10^{-5}, 2.25×10^{-5}, and 2.60×10^{-5} lb N_2/lb ClF_5-psi at 90, 120, and 150 F, respectively, were approximately 25 percent lower than those previously measured at 90 and 120 F. These measurements are being extended to higher temperature and pressure levels.

(U) The specific heat of ClF_5 was redetermined in an adiabatic calorimeter over a temperature range of 5.4 to 57.3 C (41.7 to 135 F) to resolve discrepancies in previously reported data. These data were correlated with the other experimental data over a temperature range of −70 to 57 C (−34 to 135 F) and curve fit with the following equations:

$$C_{s(cal/gm\text{-}K)} = 0.4716 - 2.217 \times 10^{-3} T_{(K)} + 8.428 \times 10^{-6} T_{(K)}^{2} - 9.453 \times 10^{-9} T_{(K)}^{3}$$

and

$$C_{s(Btu/lb\text{-}R)} = 0.4673 - 1.204 \times 10^{-3} T_{(R)} + 2.543 \times 10^{-6} T_{(R)}^{2} - 1.581 \times 10^{-9} T_{(R)}^{3}$$

This apparatus, with a new, calibrated sample chamber, is presently being used to determine the specific heat of MHF-3.

(C) Thermal conductivity measurements were completed on MHF-5 over a temperature range of 0.5 to 201.0 F. A curve fit of the data resulted in the following equation:

$$k_{(Btu/hr\text{-}ft\text{-}F)} = 0.188 - 7.91 \times 10^{-5} t_{(F)} - 2.9 \times 10^{-8} t_{(F)}^{2}$$

(U) Previously initiated saturated liquid ClF_5 viscosity measurements were extended to 75, 100, and 124 F. The resulting data, 0.323, 0.281, and 0.252 centipoises, respectively (and other data determined earlier in the program), form a plausible continuation of previously correlated low-temperature ClF_5 viscosity data. Measurements are being continued at temperatures above 200 F.

(U) As a result of the Phase III review and evaluation of the data generated under Phases I and II, efforts are continuing on the assembly of engineering property bibliographies and physical property data sheets on N_2O_4 and MMH. Unpublished data on the normal boiling point (-13.7 C or 7.3 F) of ClF_5, and ClF_5 storability in 321 stainless steel and in mild steel for periods of 31 months and 16.5 months, respectively, have been evaluated and presented. Experimental data on the heat capacity of solid ClF_3 have been curve fit with the following equations:

$$C_{p(cal/gm\text{-}K)} = -3.49 \times 10^{-2} + 3.53 \times 10^{-3} T_{(K)} - 3.07 \times 10^{-5} T_{(K)}^{2} + 1.47 \times 10^{-7} T_{(K)}^{3} - 2.57 \times 10^{-10} T_{(K)}^{4}$$

and

$$C_{p(Btu/lb\text{-}R)} = -3.49 \times 10^{-2} + 1.96 \times 10^{-3} T_{(R)} - 9.47 \times 10^{-6} T_{(R)}^{2} + 2.52 \times 10^{-8} T_{(R)}^{3} - 2.45 \times 10^{-11} T_{(R)}^{4}$$

PHASE I - LITERATURE SEARCH

OBJECTIVE

(U) The Phase I objective is the maintenance of a continuous review of the current literature and efforts of other investigators in the field to ensure acquisition and documentation of the latest possible propellant properties data for evaluation and possible inclusion into a propellant properties handbook. This survey is designed to include, but not to be necessarily limited to, the properties of the following fuels and oxidizers.

Oxidizers	Fuels
Liquid Oxygen (LO_2)	Liquid Hydrogen (LH_2)
Chlorine Pentafluoride (ClF_5)	50 N_2H_4-50 $(CH_3)_2N_2H_2$ Fuel Blend
Chlorine Trifluoride (ClF_3)	Hydrazine (N_2H_4)
Fluorine (F_2)	MMH ($CH_3N_2H_3$)
Hydrogen Peroxide (H_2O_2)	UDMH $(CH_3)_2N_2H_2$
Nitrogen Tetroxide (N_2O_4)	N_2H_4-MMH Mixtures
Mixed Oxides of Nitrogen (N_2O_4-NO)	Hybaline B-3
FLOX Mixtures (LF_2-LO_2)	Alumizine
Oxygen Difluoride (OF_2)	Pentaborane (B_5H_9)
Tetrafluorohydrazine (N_2F_4)	Diborane (B_2H_6)
	MHF Blends
	MAF Blends

RESULTS AND ACCOMPLISHMENTS

(U) A formal survey of current propellant literature, initiated under Contract AF04(611)-10546 (Ref. 1), is being continued as Phase I of the present program. This survey, which includes the location, acquisition, and documentation of all available propellant properties data of interest to the Air Force, was originally directed at a comprehensive review of physical properties data. However, under the present contract, the survey has been expanded to additional engineering properties data.

(U) This survey is being maintained through two primary techniques. The main portion of the effort is directed at the survey of all reports acquired by Rocketdyne; these include reports obtained as a result of individual personnel requests as well as those acquired through normal distribution channels. Each of these reports is surveyed with respect to subject matter, and reports containing potential propellant properties data are selected for detailed review. All pertinent data contained in these reports are documented for assembly under Phase III.

(U) To aid in the complete awareness of all available propellant properties data and their subsequent acquisition and documentation, this report survey effort is supplemented by a continuous survey of the current releases of Chemical Abstracts, NASA CSTAR Abstracts, CPIA Chemical Propulsion Abstracts, Defense Documentation Center (DDC) Technical Abstract Bulletin (TAB), NBS Cryogenic Data Center Current Awareness Service, and propellant manufacturers' bulletins. Any pertinent reports located through these sources that have not been acquired previously by Rocketdyne are ordered and reviewed in detail on their acquisition.

(U) During the current report period, 2279 reports were surveyed; of this total 118 reports were reviewed in detail for propellant properties data. The pertinent data abstracted from these reports are being compiled and evaluated under Phase III.

PHASE II: EXPERIMENTAL DETERMINATIONS

OBJECTIVE

(U) Phase II is designed for the experimental characterization of essential physical properties of selected liquid propellants. This phase essentially constitutes a 24-month continuation of the efforts initiated under Phase II of Contract AF04(611)-10746 (Ref. 1). Selection of the propellants and properties to be characterized experimentally is related to the unavailability of required data and relative importance of the data to the Air Force. Initial efforts have emphasized the completion of those propellant properties recommended for initial characterization under the previous program. Additional efforts are continuing in an order related to the importance of the data to the Air Force as determined by the Air Force Project Engineer.

(U) The selected properties are being determined over the liquidus temperature range and over a pressure range of 14.7 to 1000 psi, where practical. Changes to the selected list can be made at any time during the program through mutual agreement of Rocketdyne and the Air Force Contracting Officer. Standard test methods are being used when available. Unique or new test methods to be used must be approved by the Air Force Contracting Officer.

RESULTS AND ACCOMPLISHMENTS

(U) Phase II efforts during the current period were directed at:

1. Extension of ClF_3 phase property data into the critical region
2. Experimental characterization of sonic velocity of ClF_5 and ClF_3 at pressures above saturation

3. Continuation of $N_{2(g)}$ solubility measurements in ClF_5

4. Improvement of previously measured ClF_3 and MHF-3 specific heat data

5. Completion of MHF-5 thermal conductivity measurements

6. Extension of the ClF_5 viscosity measurements

The efforts conducted under each of these tasks are described in the following paragraphs.

Phase Property Measurements

(U) Current phase property measurements have been designed for the reliable extension of the available experimental data on ClF_3 into the critical range. Previous efforts (Ref. 2) in this program had expanded and correlated available density data (Ref. 1 and 3) on saturated liquid ClF_3 over a temperature range of -22 C (-8 F) to 161 C (322 F). Vapor pressure measurements (Ref. 2) on ClF_3 had been conducted over a temperature range of 42.9 C (109 F) to 147.6 C (298 F) to increase the reliability of previously available (Ref. 1 and 4) vapor pressure data in the high-temperature region. In addition, a critical temperature of 179.6 ±0.5 C (355.3 F) had been measured (using the liquid-vapor meniscus disappearance technique) and reported (Ref. 1).

(U) The present extension of phase property measurements into the critical region is accomplished through the experimental determination of pressure-temperature relationships of known quantities of ClF_3 using a constant-volume vapor pressure apparatus (which has been described previously in Ref. 2). To ensure attainment of reliable data, a 0- to 1000-psia range pressure transducer used with the apparatus was calibrated with a Heise gage at temperatures and pressures over the intended ranges of use. Because the calibration results showed that the transducer output was significantly sensitive to temperature changes, a second-order function was required in both the output voltage and input temperature terms during data reduction. A curve fit of the calibration data with an appropriate equation was obtained using a least-squares computer program.

(U) The technique used in the present measurements is identical to that previously used in the measurement of ClF_5 critical properties (Ref. 5). In this technique the vapor pressure apparatus is loaded with various known quantities of the liquid. As the temperature of each liquid sample is increased in increments (through regulated external heating of the apparatus in a Fisher Isotemp oven), the pressure of the sample is recorded (after the sample has reached thermal equilibrium). This pressure represents the vapor pressure of the sample as long as both the liquid and vapor phases of the sample are still present. The temperature (discontinuity temperature) at which a change in slope is observed from the vapor pressure curve (as noted in a plot of log pressure vs reciprocal absolute temperature) marks the transition of the sample from a liquid-vapor system to a single-phase system through complete vaporization or liquid expansion. The critical point can be determined from these data by location of the maximum discontinuity temperature in a plot of the temperatures of discontinuity (for the various sample sizes) vs the sample size, or the specific volume or density of the sample at the transition.

(U) The discontinuity temperature plot will provide the temperature, specific volume, and density of ClF_3 at the critical point. The vapor pressure measurements required to establish this plot will establish a vapor pressure curve for this region from which the critical pressure can be calculated (at the critical temperature). In addition, the measurements will provide saturated liquid or vapor densities at each of the transition temperatures.

(U) Chlorine Trifluoride Vapor Pressure. During the current report, two series of pressure-temperature measurements (each using a different known amount of propellant-grade ClF_3) were completed. The vapor pressure data resulting from these measurements are given in Table 1 as runs 2 and 3. In addition, previously reported vapor pressure data (Ref. 2) have been corrected on the basis of new instrument calibration data and are reported in Table 1 as run 1. These preliminary data were curve fit from 25.65 to 148.58 C (78.17 to 299.44 F) with the following equations:

$$\log P_{(psia)} = 5.9929 - \frac{2453.7}{T_{(R)}} \qquad (1)$$

and

$$\log P_{(atm)} = 4.8257 - \frac{1363.1}{T_{(K)}} \qquad (2)$$

(U) Chlorine Trifluoride Critical Properties. Additional pressure-temperature data obtained during the above vapor pressure measurements have been used to establish three temperatures of discontinuity from Eq. 1. Although measurements are being continued on several more sample sizes to establish reliable ClF_3 critical property and high-temperature vapor pressure data, an estimation of ClF_3 critical properties has been made from the above data. Using the previously determined (Ref. 1) critical temperature of 179.6 ±0.5 C (355.3 F), the extension of the above vapor pressure equation would result in a calculated critical pressure of 961 psia (65.4 atm). Extrapolation of the reported (Ref. 2) density data and the presently available temperature of discontinuity values to the above critical temperature results in a ClF_3 critical density of 0.88 gm/cc (54.9 lb/cu ft) and a specific volume of 1.14 cc/gm (0.018 cu ft/lb).

TABLE 1

PRELIMINARY CHLORINE TRIFLUORIDE VAPOR PRESSURE DATA

Run No.	Temperature		Vapor Pressure, psia
	C	F	
2	25.65	78.17	25.4
1	42.90	109.22	46.3
3	47.43	117.37	58.4
2	48.15	118.67	56.4
1	61.10	141.98	74.3
2	70.75	159.35	106.9
1	74.58	166.24	112.7
3	81.30	178.34	130.4
1	93.13	199.63	180.1
2	96.75	206.15	202.9
3	107.70	225.86	279.0
1	111.30	232.34	271.6
2	121.00	249.80	341.4
1	131.35	268.43	404.7
2	133.05	271.49	457.4
3	148.03	298.45	614.5
1	148.28	298.90	559.1
1	148.58	299.44	556.7

Sonic Velocity (and Compressibility) Measurements

(U) Determinations of sonic velocity and adiabatic compressibility of liquid propellants are being conducted with the apparatus described in detail in Ref. 2. With this apperatus the velocity of sound in a liquid is obtained experimentally by measuring the time required for an acoustic wave to travel a known distance in the test liquid. Using the resulting sonic velocity data and the liquid density, the adiabatic compressibility of the liquid is calculated from the relationship:

$$\beta_s = \frac{1}{\rho c^2}$$

where

β_s = adiabatic compressibility of the liquid

ρ = density of the liquid

c = velocity of sound in the liquid

(U) During previous sonic velocity measurements (Ref. 2) in ClF_5 and ClF_3 at saturated liquid conditions and under total pressurization (with gaseous nitrogen) of 500 and 1000 psia, anomalies were noted in the data resulting from the pressurized liquid measurements. Normally, the velocity of sound in a liquid at constant temperature has been found to increase with increasing pressure. In addition, the change in slope of a sonic velocity-temperature plot is relatively equivalent for various isobars over the same temperature range. However, the data obtained at the 500- and 1000-psia levels for both ClF_5 and ClF_3 indicated that at the lower temperatures investigated, the velocity of sound was decreased as the pressure was increased and that these conditions were reversed as temperature was increased.

(U) Since the sonic velocities in both ClF_5 and ClF_3 were reliably established for the saturated liquid conditions (Ref. 2), it was postulated that the behavior under the pressurized conditions was an effect of pressurant gas solubility in the liquid. To evaluate this hypothesis and correctly determine sonic velocities in compressed liquids (in the absence of such effects), the experimental technique was altered. In this modification, the liquid sample in the experimental apparatus was thermostated at a selected temperature and pressurized to the desired total pressure level with gaseous nitrogen. After the immediate determination of sonic velocity, the sample was depressurized, the nitrogen removed (through use of an expansion and cold-trap process), and the technique was repeated at a new temperature and/or pressure level.

(U) Sonic Velocity in Chlorine Pentafluoride. As a result of the effort to demonstrate and reduce the effect of the dissolved pressurant gas, sonic velocity measurements were repeated in ClF_5 under pressurized conditions of 500, 800, and 1000 psia using the modified procedure. The data from these measurements, which are compared in Table 2 with the data previously established (Ref. 2) for the saturated liquid, substantiate the hypothesis of the previous effect (Ref. 2) of the pressurant gas and demonstrate the expected behavior in the absence of such an effect. These data are presently being curve fit for graphical representation in the next quarterly technical report.

(U) Sonic Velocity in Chlorine Trifluoride. Sonic velocity measurements were also repeated (using the technique modification) in ClF_3 under total pressurizations of 500, 800, and 1000 psia. The data from initial measurements at -14.12 C (6.6 F) and 4.43 C (40.0 F) are shown in Table 2 with the previously established (Ref. 2) saturated liquid data. After additional measurements are conducted at higher temperatures, the data will be evaluated and curve fit.

TABLE 2

VELOCITY OF SOUND IN LIQUID
CHLORINE PENTAFLUORIDE AND CHLORINE TRIFLUORIDE

Liquid	Temperature		Pressure,	Sonic Velocity	
	C	F	psia	m/sec	ft/sec
ClF_5	-14.78	5.4	Saturation	742.0	2434.4
			500	755.8	2479.7
			800	765.6	2511.8
			1000	770.9	2529.2
	2.42	36.4	Saturation	677.4	2222.4
			500	691.1	2267.4
			800	700.6	2298.5
			1000	707.1	2319.9
	23.50	74.3	Saturation	598.7	1964.2
			500	616.1	2021.3
	45.88	112.8	Saturation	515.7	1691.9
			500	535.0	1755.2
			1000	558.4	1832.0
	69.20	156.6	500	449.0	1473.1
ClF_3	-14.12	6.6	Saturation	991.9	3254.3
			500	1002.6	3289.4
			800	1010.0	3313.6
			1000	1013.5	3325.1
	4.45	40.0	Saturation	923.1	3028.5
			500	933.5	3062.7
			800	939.2	3081.4
			1000	945.1	3100.7

Inert-Gas Solubility Measurements

(U) Measurements of inert-gas solubility in liquid propellants are being conducted in an apparatus which has been previously decribed in detail (Ref. 1,2). In this apparatus, the inert gas is introduced from a volume-calibrated gas reservoir into a volume-calibrated test tank, or propellant cell, which contains a known quantity of propellant. The volume of the gas absorbed at a known temperature and after agitation is calculated from pressure changes that occur in the system. These pressure changes are monitored by two precision differential pressure transducers. The entire apparatus, including both the propellant cell and the gas reservoir, is mounted within a thermostated enclosure, which maintains a desired temperature during a solubility determination. The temperature-conditioning enclosure is supported upon a rocking platform which is used to agitate the test solution in the propellant cell to attain equilibrium conditions.

(U) In these determinations, the experimentally derived quantity is a differential solubility (the gas dissolved per unit mass of propellant and per unit pressure increase) measured at a particular temperature and pressure. This quantity can readily be integrated to give total (absolute) solubility.

(U) Previous use of this apparatus in this program resulted in preliminary determinations of nitrogen gas solubility in ClF_5 at 90 and 120 F (Ref. 2). During these original measurements, the solubility estimate was based on the establishment of two successive pressure equilibria in the propellant cell: (1) after introduction of the gas into the ullage of the propellant cell (but before appreciable solution could occur),and (2) after agitation of the cell contents to induce solution equilibrium. However, this approach has proved impractical with the present apparatus because presumably, the gas input causes propellant vapor condensation, and slow diffusion-controlled re-evaporation leads to false, unrelated equilibria at both stages of the process (Ref. 2).

(U) To avoid this misinterpretation of the experimental results, present solubility measurements are being conducted with a variation of the original experimental procedure. In this modified procedure, the two basic steps (pressurization of the ullage without solution, and pressurization of the cell with solution) are evaluated independently. Each of these two pressure responses is determined from a separate sequence of gas inputs (without and with cell agitation, respectively), which deliberately set up a reproducible vapor-gas, pseudo equilibrium in the ullage for each sequence. The eventual consistency of pressure responses for each sequence during subsequent experimentation substantiates the assumptions made.

(U) Nitrogen Gas Solubility in Chlorine Pentafluoride. During the current report period, measurements of gaseous nitrogen solubility in ClF_5 were conducted at 90, 120, and 150 F through the use of the newly modified technique. These data (Table 3) are believed reliable within 3 or 4 percent (contingent upon additional experience with the technique being utilized). The values shown for measurements at 90 and 120 F are significantly lower (by ~25 percent) than the values previously reported (Ref. 2); however, the new data are preferred due to the previous experimental difficulties and the subsequent problem of data interpretation.

TABLE 3

NITROGEN GAS SOLUBILITY IN CHLORINE PENTAFLUORIDE

Temperature, F	ClF_5 Vapor Pressure, psia	Total Pressure, psia	Solubility, lb N_2/lb ClF_5-psi
90	72	320 to 470	1.83×10^{-5}
120	115	580 to 620	2.25×10^{-5}
150	176	740 to 780	2.60×10^{-5}

(U) Although the recent data obtained are apparently reliable, additional experience in the use of the new technique could indicate defects in this approach as well. In any case, the technique is extremely laborious and, therefore, somewhat impractical in providing data for a large number of propellants. Thus, to accomplish the overall objective of this effort, improvements may be sought through further modifications of the apparatus. Presently, nitrogen gas solubility determinations are being conducted in ClF_5 at higher temperatures and at different pressure levels; the latter determinations should indicate any significant deviations from constant solubility (Henry's Law) behavior.

Specific Heat Measurements

(U) Experimental determinations of liquid propellant specific heats are being conducted in a calorimeter previously developed and described under Contract AF04(611)-9563 (Ref. 5) and further described and utilized under Contract AF04(611)-10546 (Ref. 1) and in the present program (Ref. 2). During the current report period, efforts under the specific heat measurement task were directed at: (1) investigation and correction of the poor precision and apparently anomalous results of preliminary specific heat measurements on the MHF-3 fuel blend, and (2) resolution of the discrepancy in previously reported ClF_5 specific heat values.

(U) An explanation was sought for the large scatter (up to 5 percent) in the MHF-3 specific heat data reported in Ref. 2. The sample container employed in the initial MHF-3 measurements was recalibrated empty under carefully controlled conditions at 0 C. Since the results indicated that the system was extremely sensitive to small temperature gradients, an electrically heated adiabatic shield has been installed in place of the glass dewar (described in Ref. 2) which had been used to minimize temperature gradients in the calorimeter. Also, the sample container was suspended in the center of the heat shield by cotton thread attached to the heat shield lid.

(U) In this modification, a differential thermocouple is used to monitor and nullify the temperature difference between the shield and the sample container. Electrical interaction between the differential thermocouple circuit and the sample thermocouple is prevented by insertion of a thin piece of lens paper between the differential thermocouple junctions and the walls they contact. The signal from this thermocouple is fed into a d-c microvolt amplifier and then to a recorder and a current control unit (which continuously feeds the necessary power into the shield by means of a silicon-controlled rectifier power supply unit). The controller is set to regulate the shield temperature at some small temperature increment above that of the sample container to compensate for heat losses through the container leads to the heat sink (which is provided by the controlled-temperature bath in which the apparatus is immersed). The K-3 potentiometers, used to determine the energy supplied to the heaters and the sample container thermocouple output, have been provided with new Eppley standard cells, and the 6-volt batteries used to heat the sample container have been replaced with a constant-current power source.

(U) During calibrations of a sample container for MHF-3, it was necessary to employ two calorimeter bath media (dry ice-methanol and ice water) as constant-temperature heat sinks to span the desired temperature range. However, small leaks in the calorimeter O-ring seal, which occurred while the calorimeter was immersed in the dry ice-methanol bath, has led to replacement of the O-ring with a specially fabricated Teflon gasket. This replacement has sustained a good vacuum ($\sim 10^{-5}$ mm Hg) at low temperatures.

(U) <u>Specific Heat of Saturated Liquid Chlorine Trifluoride</u>. Because of continuing requirements for accurate ClF_3 specific heat data over extended temperature ranges, a decision was made to resolve the existing discrepancy previously reported (Ref. 1) in the data from two different experimenters (Ref. 1 and 4). For this purpose, a new sample container was fabricated of copper with a re-entrant well and four fins were brazed (with silver solder) lengthwise to the outer wall. The bottom plate was constructed of stainless steel to restrict thermal conduction from the outer wall across the bottom to the re-entrant well. The heater winding of No. 30 B&S-gage constantan wire was noninductively wound on the sample container and coated with GE varnish No. 7031. While the varnish was still tacky, an outer sheet of copper foil was attached.

(U) The new sample container (with a volume of ~59 cc) was calibrated in the adiabatic calorimeter from 3.5 to 56.3 C (38.3 to 133.3 F). During this calibration, in which the heat capacity contribution of the sample container was determined, the container was loaded with ~0.05 atm helium gas (negligible heat capacity contribution) for thermal equilibration. The deviation of the 15 individual data values from the correlated fit of the data was quite small (≤0.5 percent).

(U) After passivation of the sample container, 97.37 grams of propellant-grade ClF_3 was condensed into the sample container from a vacuum line; the fill line was crimped and sealed with soft solder. The specific heat of this saturated liquid sample was measured over a temperature range of 5.4 to 57.3 C (41.7 to 135 F). The resulting data, which are listed in Table 4 with the chemical analysis of the ClF_3 sample, indicate excellent agreement with the extension of the saturated liquid ClF_3 specific heat data reported in Ref. 4 (from the melting point to the normal boiling point). As a result, the two sets of data were curve fit from -70 to 57 C (-34 to 135 F) with the following equations:

$$C_{s(cal/gm\text{-}K)} = 0.4716 - 2.217 \times 10^{-3} T_{(K)} + 8.428 \times 10^{-6} T_{(K)}^{2} - 9.453 \times 10^{-9} T_{(K)}^{3}$$

TABLE 4

EXPERIMENTAL SPECIFIC HEAT OF
SATURATED LIQUID CHLORINE TRIFLUORIDE*

Temperature C	Temperature F	Specific Heat, cal/gm-C
5.38	41.68	0.306
6.29	43.32	0.309
8.72	47.70	0.301
9.09	48.36	0.305
11.14	52.05	0.306
14.36	57.85	0.306
17.17	62.91	0.307
19.63	67.33	0.304
20.05	68.09	0.307
22.20	71.96	0.312
24.76	76.57	0.307
24.98	76.96	0.306
26.55	79.79	0.311
27.62	81.72	0.310
28.49	83.28	0.311
31.07	87.93	0.312
31.50	88.70	0.310
34.67	94.41	0.313
38.19	100.74	0.312
42.04	107.67	0.316
45.85	114.53	0.316
49.68	121.42	0.317
53.49	128.28	0.317
57.30	135.14	0.317

*Sample Composition:

ClF_3, w/o	99.0
$FClO_2/ClO_2$, w/o	0.88
HF, w/o	0.11
ClF	trace

and

$$C_{s(Btu/lb\text{-}R)} = 0.4673 - 1.204 \times 10^{-3} T_{(R)} + 2.543 \times 10^{-6} T_{(R)}^{2} - 1.581 \times 10^{-9} T_{(R)}^{3}$$

A graphical representation of these data are shown in Fig. 1 with the heat capacity of solid ClF_3 (noted as a result of Phase III efforts).

(C) Specific Heat of MHF-3. Because the ClF_3 specific heat measurements provided an additional indication that the adiabatic calorimeter was functioning properly, specific heat measurements were continued on the MHF-3 fuel blend (nominal composition: 86 w/o $CH_3N_2H_3$-14 w/o N_2H_4). A new sample container, similar in construction to the one used for ClF_3, was fabricated and leak-checked in preparation for its use with MHF-3 and other similar fuel blends. Calibration heat capacity measurements were completed successfully with this sample container (loaded with ~ 0.05 atm He) inserted in the calorimeter. Values obtained from two separate calibration runs, which were necessitated by the replacement of an O-ring in the apparatus with a Teflon gasket, resulted in a linear heat capacity-temperature curve from -50.1 to 61.6 (-58.2 to 142.9 F); the average point deviation from the smoothed curve was ±0.5 percent.

(U) The sample container was loaded (under nitrogen in a glove bag) with 58.43 grams of MHF-3 fuel blend. After the filling tube of the sample container was crimped and brazed with silver solder, the calorimeter was reassembled; specific heat measurements on MHF-3 are continuing.

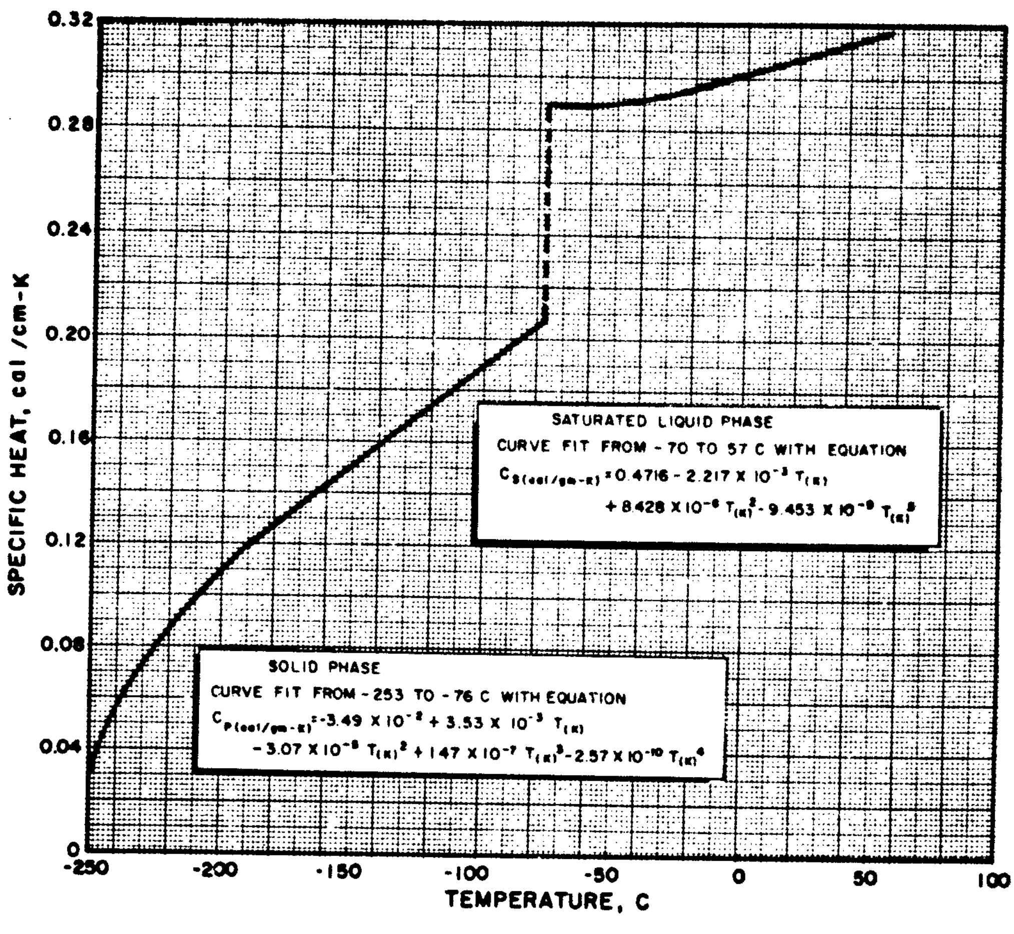

Figure 1. Specific Heat of Solid and Saturated Liquid Chlorine Trifluoride

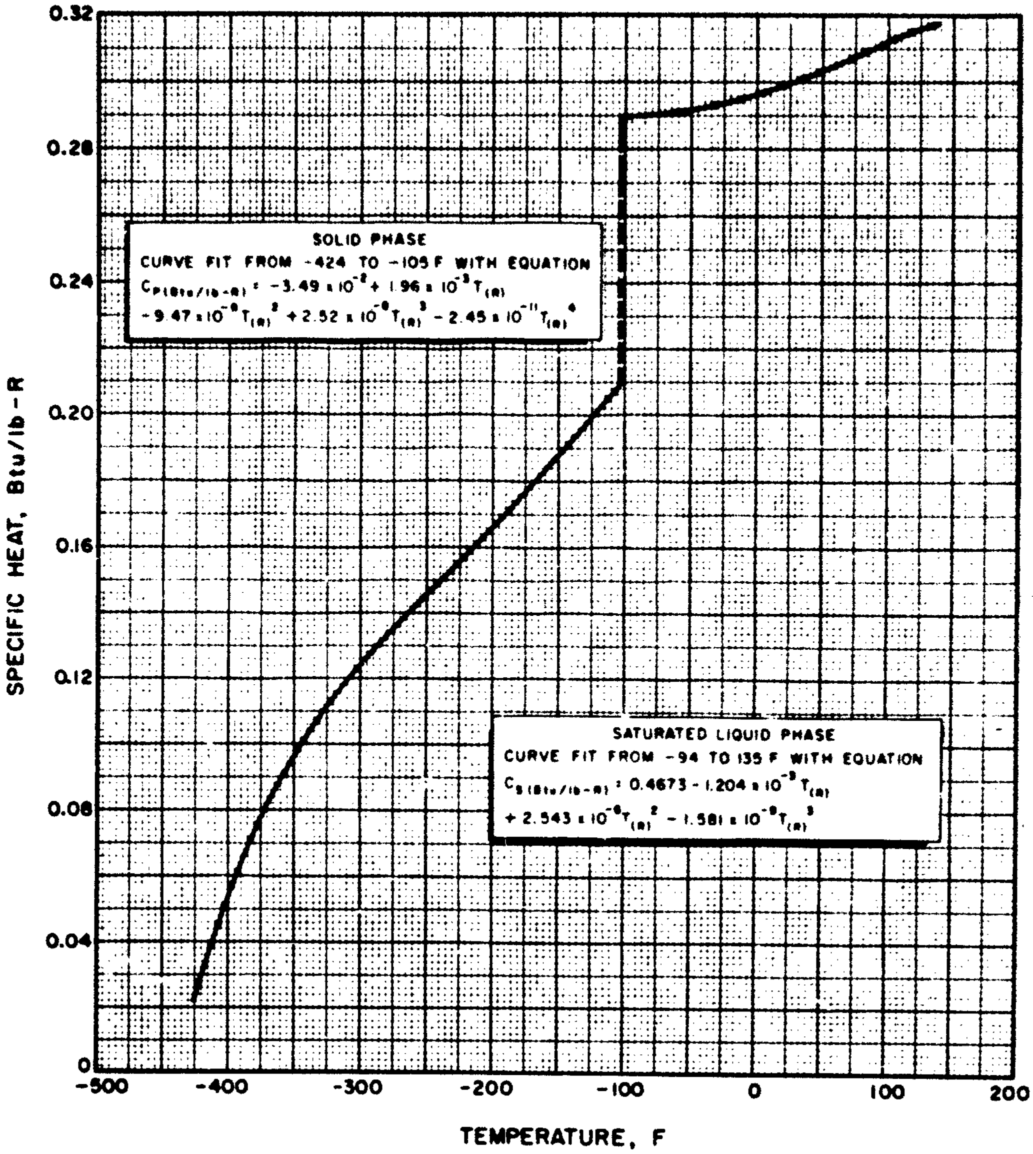

Figure 1. (Concluded)

Thermal Conductivity Measurements

(U) Thermal conductivity measurements of liquid propellants are being conducted through the use of a steady-state, concentric-cylinder conductivity cell described previously in Ref. 1 and 2. With this apparatus the test liquid is contained in a very thin, annular passage between two aluminum alloy cylinders. An electrical resistance heater, located at the center of the inner cylinder, supplies the heat energy to establish a temperature gradient across the liquid layer. Copper constantan thermocouples, embedded in cylinders close to the surfaces of the test fluid cavity, are used to measure the temperature gradient. A regulated d-c power supply is employed for the cell heater power.

(C) Thermal Conductivity of MHF-5. During the current report period, thermal conductivity measurements were completed on the MHF-5 fuel blend (nominal composition: 55 w/o $CH_3N_2H_3$-26 w/o N_2H_4-19 w/o $N_2H_5NO_3$). Earlier in the program (Ref. 2), an initial series of measurements was made on one sample (A) of the fuel blend at approximately 50 F intervals over a temperature range of 0.5 to 201.0 F. To increase the reliability of these initial data, a series of measurements was made on a second sample (B) of the propellant over approximately the same temperature range. The results from both series of measurements, which indicate good agreement with each other, are listed in Table 5.

(C) Two unsuccessful attempts were made to obtain MHF-5 thermal conductivity data at temperatures above 200 F. With sample A in the cell, an attempt was made to obtain data at 250 F. As the cell was being heated from 200 to 250 F, gas evolution was observed when the cell temperature reached approximately 225 F; at this point the test run was terminated. A similar situation occurred with sample B in the thermal conductivity cell. After data were obtained at 200 F, the cell was allowed to remain at this temperature. Approximately 2-1/2 hours later, some gas evolution was observed and the test run was terminated.

TABLE 5

EXPERIMENTAL MHF-5 THERMAL CONDUCTIVITY DATA

Sample	Temperature, F	Thermal Conductivity, Btu/hr-ft-F
A*	0.5	0.186
A	0.5	0.188
B*	0.5	0.188
B	0.5	0.189
A	50.9	0.185
A	50.8	0.185
B	50.8	0.186
B	50.8	0.184
A	100.6	0.180
A	100.6	0.180
B	100.6	0.178
B	100.6	0.179
A	150.5	0.175
A	150.5	0.176
B	150.6	0.175
B	150.6	0.176
A	200.8	0.172
A	201.0	0.171
B	200.5	0.172
B	200.5	0.170

*Sample Composition:

$CH_3N_2H_3$, w/o	54.9
N_2H_4, w/o	25.4
$N_2H_4 \cdot HNO_3$, w/o	18.9
NH_3, w/o	0.2
H_2O, w/o	0.6

(C) The thermal conductivity data were curve fit from 0.5 to 201.0 F (Fig. 2). The equation which represents the data is

$$k_{(Btu\ hr-ft-F)} = 0.188 - 7.91 \times 10^{-5} t_{(F)} - 2.9 \times 10^{-8} t_{(F)}^2$$

(U) Both propellant samples (A and B) were obtained from a specific batch of propellant blended for the thermal conductivity measurements. Results of chemical analysis of the fuel blend are given in Table 5.

Viscosity Measurements

(U) Viscosity measurements on liquid propellants are being conducted in an apparatus, shown schematically in Fig. 3, using a technique previously described in Ref. 2. In this apparatus, the viscosity is obtained by observing the flowrate through the the capillary tubing and the corresponding driving fluid head, which in this apparatus is a simple gravity head resulting from a difference in the elevation of the liquid level in the two legs of the U-tube. The reservoir in one of these legs is a section of 0.75-inch tubing which contains a magnetic float at the gas-liquid interface. The position of this float within a vertical range of approximately 6 inches is sensed by a differential transformer unit surrounding the tubing. For the other leg of the U-tube, valves B and C provide a choice between a 1-1/2-inch and 3/8-inch tubing reservoir.

(U) The viscometer is constructed entirely of stainless-steel tubing and fittings to permit testing corrosive liquids at pressures up to 1000 psi. The capillary tubing is 0.023-inch ID and approximately 26-1/2 inches long. This long, large-bore capillary was selected in an attempt to minimize the relative importance of entrance and exit friction losses at low kinematic viscosities, which may approach 0.1 centistoke for the propellants of interest. For the case of flow between the

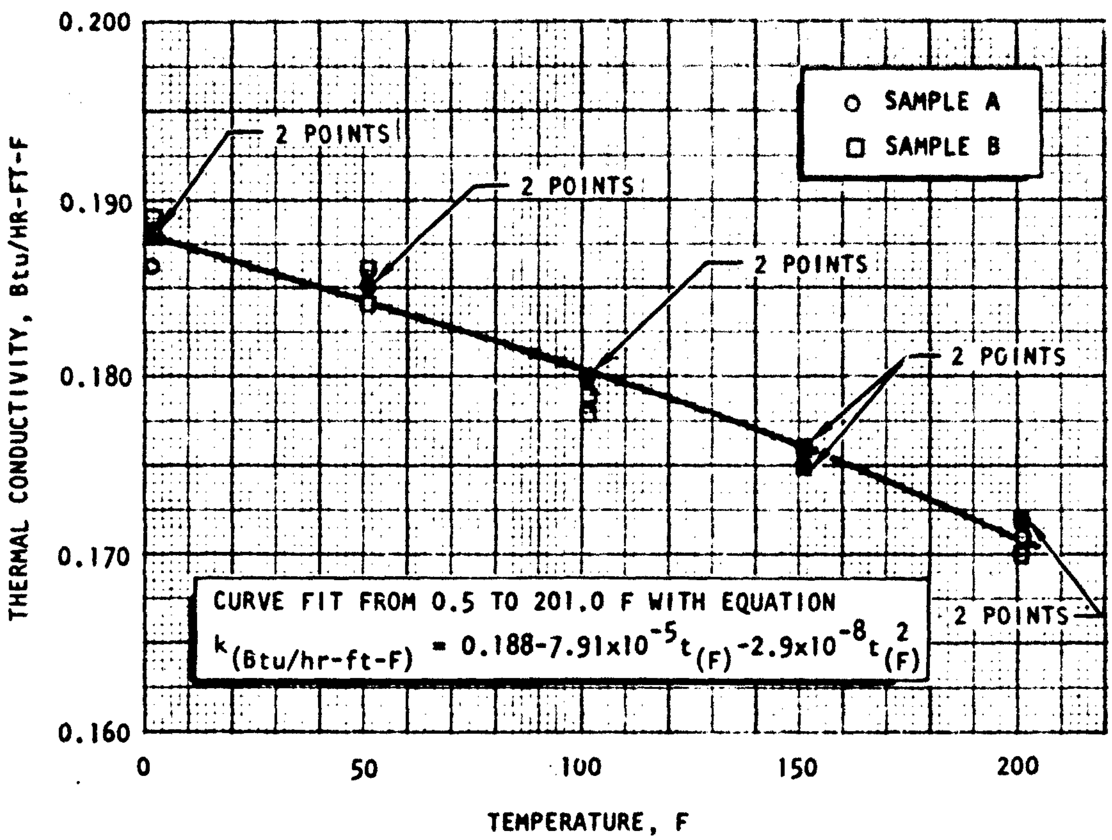

Figure 2. Thermal Conductivity of MHF-5

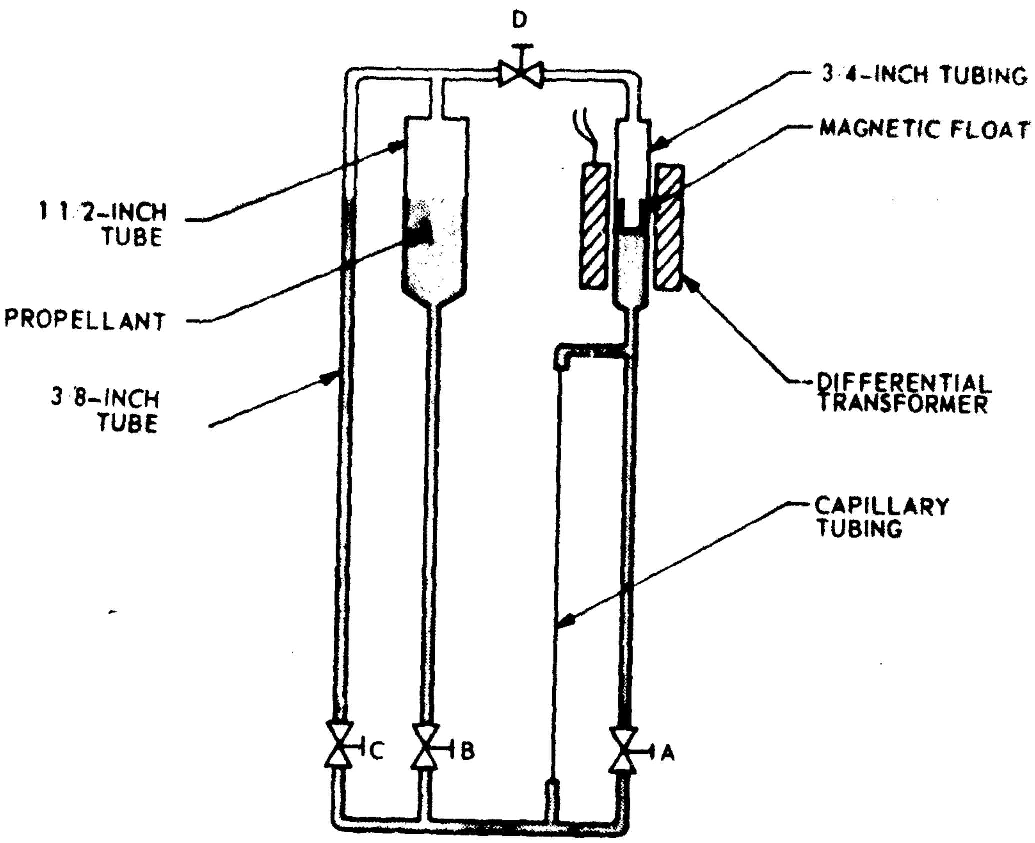

Figure 3. Capillary Viscometer Schematic

3/4-inch float reservoir and the 1-1/2-inch reservoir, this capillary would lead to experimental flow times ranging from minutes to hours in the viscosity range contemplated. To increase the speed of the test process at the higher viscosities, the option of the 3/8-inch reservoir has been provided, which permits setting up a much larger differential head within the operating range of the transducer and float. In addition, this option permits testing of a given viscosity under significantly different driving heads, flow velocities, and hence, Reynolds numbers, for the capillary. This feature could permit estimation or calibration of the end effects at low viscosities. It may be possible to extend the applicability of the viscometer into a range where end effects are important through a technique of self-calibration in which a given viscosity is measured over a sufficiently wide Reynolds number range.

(U) Not shown in the figure are accessory connections and valving for loading, venting and/or pressurizing the viscometer with inert gas. The overall unit, approximately 5 feet tall, is housed in a temperature-controlled dry box equipped with heater, circulation fans, thermocouples, pressure transducer, etc.

(U) Viscosity of Chlorine Pentafluoride. Viscosity measurements in ClF_5, which were initiated previously in this program (Ref. 2), have been continued at 75, 100, and 124 F. These measurements have been designed to extend the previously reported saturated liquid ClF_5 viscosity data (Ref. 5 and 6) to higher temperature and pressure ranges. The results from the current measurements are shown (with those obtained earlier) in Table 6.

TABLE 6

EXPERIMENTAL CHLORINE PENTAFLUORIDE VISCOSITY DATA

Temperature, F	Temperature, C	Kinematic Viscosity, centistokes	Absolute Viscosity, centipoises
75	24	0.182	0.323
100	38	0.164	0.281
124	51	0.151	0.252
154*	68	0.134	0.215
176*	80	0.121	0.185

*Data obtained earlier in the program (Ref. 2)

(U) Each of the indicated data points represents at least three measurements within ±1 F and 1-percent precision. Density data for the reduction of kinematic viscosity to absolute viscosity was taken from Ref. 5. Figure 4 compares the data shown in the table with a correlation (Ref. 1) of viscosities previously obtained at lower temperatures in conventional glass viscometers (Ref. 5 and 6). As shown, the new higher-temperature values form a smooth and plausible continuation of the early data although the curve fit may require some modification. An extrapolation of the curve fit of the lower-temperature data, through the temperature range of the present measurements, is indicated by the dashed line.

(U) The viscometer is presently being readied for measurements above 200 F to provide additional high-temperature data on ClF_5 and test the low-viscosity applicability of the capillary.

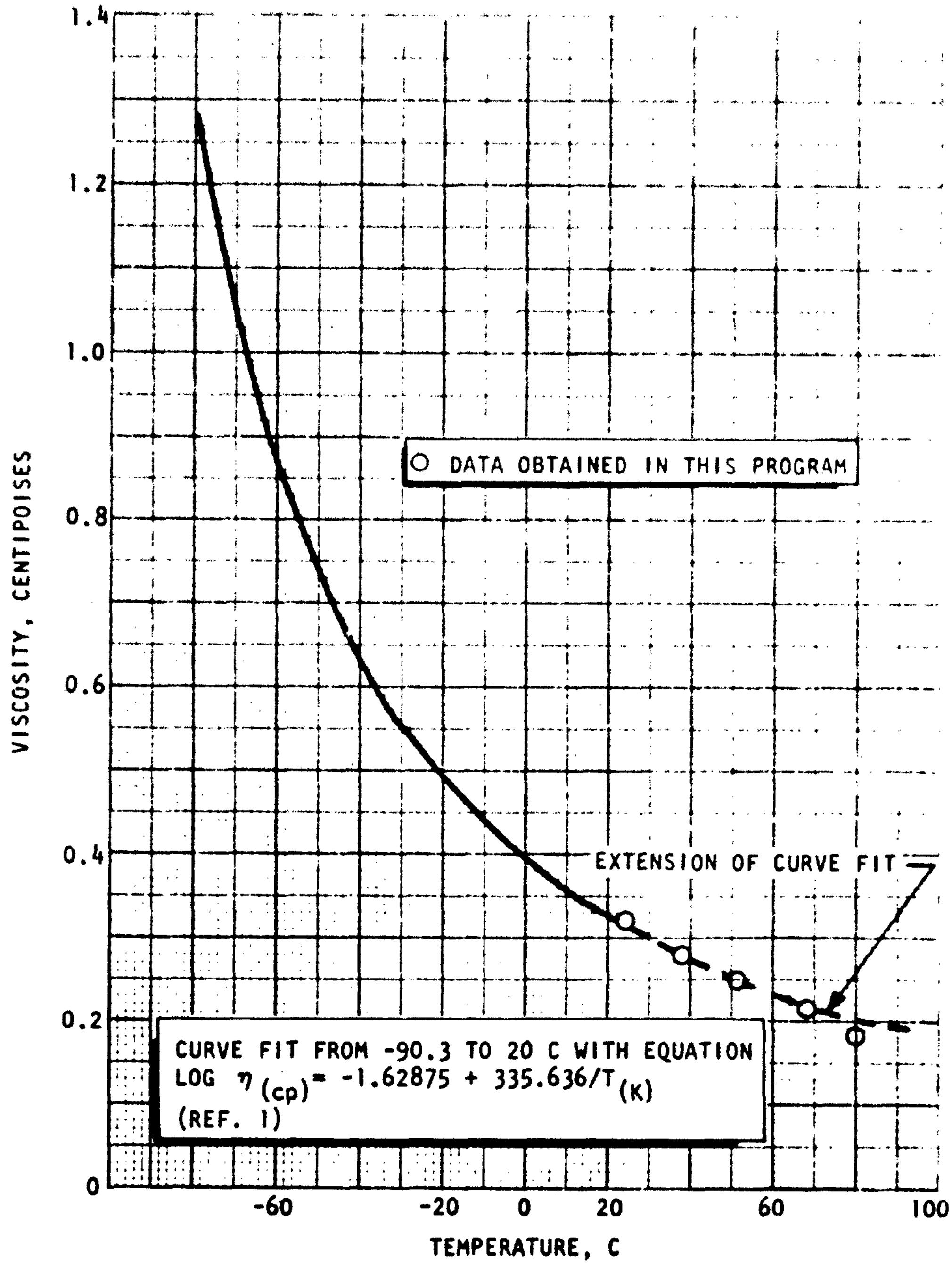

Figure 4. Viscosity of Saturated Liquid Chlorine Pentafluoride

PHASE III: EVALUATION AND COMPILATION OF DATA

OBJECTIVE

(U) During the entire period of the contractual program, efforts under Phase III are directed toward the assembly of all data generated by Phases I and II, verification of all the data sources, critical comparison of conflicting data, and tabulation of the results. The final selection of the best values will be summarized in an annual technical report.

RESULTS AND ACCOMPLISHMENTS

(U) In the current report period, Phase III effort has been directed primarily at the compilation and evaluation of physical properties of various selected propellants from the references located in the Phase I literature survey. The remaining effort has been concentrated on the reduction, evaluation, and correlation of all data generated as a result of the Phase II experimental efforts; however, to maintain continuity in this text, these results are reported under the pertinent Phase II headings. In addition, unpublished physical and engineering property data on propellants (generated as a result of Rocketdyne in-house efforts) which are related to the overall objectives of this program are given in the following text.

Nitrogen Tetroxide Property Compilations

(U) As a result of the comprehensive search of the propellant properties literature, all pertinent engineering property data for N_2O_4 is being compiled, evaluated, and correlated. The references obtained from this search, which are being placed on indexed punch cards, are being used to prepare an engineering properties bibliography. The physical property data are being summarized for future publication under this program.

Monomethylhydrazine Property Compilations

(U) Compilations of an engineering properties bibliography and physical properties data sheets for MMH, $CH_3N_2H_3$, have been initiated.

Normal Boiling Point of Chlorine Pentafluoride

(U) The normal boiling point of ClF_5 was experimentally measured (during a Rocketdyne in-house effort) as -13.7 C (7.3 F). These measurements were conducted using a calibrated copper constantan thermocouple to indicate the equilibrium temperature when the equilibrium vapor pressure (as determined by a Heise gage) was 760 mm Hg. The ClF_5 sample used in the measurements contained fused cesium fluoride at the bottom of the sample to complex any HF impurity and thus minimize the contribution of the more volatile impurity. Chemical analysis of the sample indicated an assay > 99.6 w/o ClF_5.

Heat Capacity of Chlorine Trifluoride (Solid)

(U) In conjunction with the evaluation of the experimental data on the specific heat of saturated liquid ClF_3 (as noted in Phase II), the experimental heat capacity data on solid ClF_3 were also evaluated. These data, which were determined from 14.04 K (-259.12 C or -434.4 F) to the melting point by the same experimentalists (Ref. 4) who provided the original specific heat data on the saturated liquid, were curve fit with the following equations:

$$
\begin{aligned}
C_{p(cal/gm\text{-}K)} = & -3.49 \times 10^{-2} + 3.53 \times 10^{-3} T_{(K)} \\
& -3.07 \times 10^{-5} T_{(K)}^{2} + 1.47 \times 10^{-7} T_{(K)}^{3} \\
& -2.57 \times 10^{-10} T_{(K)}^{4}
\end{aligned}
$$

and

$$C_{p(Btu/lb\text{-}R)} = -3.49 \times 10^{-2} + 1.96 \times 10^{-3} T_{(R)}$$
$$-9.57 \times 10^{-6} T_{(R)}^{2} + 2.52 \times 10^{-8} T_{(R)}^{3}$$
$$-2.45 \times 10^{-11} T_{(R)}^{4}$$

These data are also presented graphically in Fig. 1.

Storability of Chlorine Pentafluoride

(C) Storability tests on ClF_5, which were completed over a 13-month period under Contract AF04(611)-9563 (Ref. 7), were extended over a longer period under Rocketdyne funding. In the program conducted under the contract, liquid samples of ClF_5 were stored in 321 stainless steel, 6061 aluminum, Monel 400, and oxygen-free copper containers (initial ullages of ~20 to 30 percent) for 13 months at ambient temperatures. In addition to weekly monitoring of container pressures and ambient temperatures, liquid and vapor samp'es were removed from each container and chemically analyzed monthly. The results, which are given in detail in Ref. 7, indicated no significant changes in pressure or composition during the entire storage period.

(C) In an extension of these tests (initiated on 12 November 1964), the storage period of the 321 stainless-steel container was continued to 22 June 1967, which represented 31 months of storage at temperatures ranging from ~30 to 100 F. The final chemical analysis of the liquid sample from this container was as follows:

ClF_5, w/o	99.4
ClF_3, w/o	0.3
HF, w/o	0.3

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This analysis indicates no significant change from the original analysis of the liquid sample which was noted in Ref. 7 as:

ClF_5, w/o	99.1
ClF_3, w/o	<0.4
ClF, w/o	<0.4
$F_2 \cdot CF_4$, w/o	<0.03
HF, w/o	0.4

The small differences reported in the assays and in the noted impurities between the two analyses are attributable to uncertainties in the original analytical technique and subsequent improvements.

(C) In addition to the results from the controlled storage of ClF_5, an evaluation was conducted on the storage of ClF_5 in a typical open-hearth steel shipping cylinder (ICC-3AA2400). This evaluation was made on a shipping cylinder that had been loaded with 133 pounds of ClF_5 on 16 February 1966, shipped to Dahlgren, Va., where 93 pounds were removed, and returned to Rocketdyne with 43 pounds of ClF_5. The results of chemical analysis of the cylinder contents after loading (16 February 1966) and on 3 July 1966 (after its return to Rocketdyne) are compared as follows:

Composition	Analysis, w/o	
	16 February 1966	3 July 1966
ClF_5	99.3	99.6
HF	<0.5	0.4
Cl	<0.1	
Cl_2	<0.01	
ClO_2	<0.01	
ClF_3	<0.1	
$FClO_3$	<0.01	

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[redacted]

These results indicate that there were no significant composition changes in the ClF_5 during the 16.5-month storage in the particular shipping cylinder. As noted previously, the slight differences in the analytical results are a result of improvements in the analytical technique since the initial analysis was conducted.

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13 ABSTRACT

The results of the fifth quarter year's effort of a 24-month program on the analytical and experimental characterization of the physical properties of selected liquid propellants are presented in three phases. In Phase, I, a continuous review of the literature was conducted to ensure the acquisition and documentation of the latest possible propellant properties data for evaluation and possible inclusion into a propellant properties handbook. Phase II experimental efforts have resulted in the measurement of chlorine trifluoride vapor pressure in the high-temperature region and an estimation of critical properties; sonic velocity in chlorine trifluoride and chlorine pentafluoride under pressurized conditions; nitrogen gas solubility in chlorine pentafluoride; specific heat of saturated liquid chlorine trifluoride; thermal conductivity of MHF-5; and viscosity of chlorine pentafluoride. Phase III efforts included the evaluation and assembly of all data generated in Phases I and II, a curve fit of specific heat data on solid chlorine trifluoride, and a report of unpublished data on the normal boiling point of chlorine pentafluoride and chlorine pentafluoride storability. (U)

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